



A Holding Time Evaluation Study for the Analysis of PFAS in Aqueous Samples

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Per/Polyfluorinated Compounds



- Per/Polyfluorinated Alkyl Substances (PFAS) are a class of compounds that have been in use since the late 1940's, early 1950's.
- PFAS compounds, in general, are persistent in the environment and bioaccumulative.
- Because of that we might expect that persistence and stability to impact holding times referenced in analytical methods.
- Persistent organic pollutants (POPs) like PCBs and Dioxins/furans reference holding times of 1 year.

PFAS Holding Times



- EPA Method 537 ver 1.1 stipulates a 14 day holding time, when using polypropylene containers and Trizma as a preservative
- ISO 25101 uses a 14 day holding time with polypropylene and sodium thiosulfate as preservative
- ASTM D7979-17 uses a 28 day holding time with a polypropylene tube and dilution with methanol
- DoD/QSM 5.1.1 using a 14 day holding time with high density polyethylene (HDPE) containers

PFAS Holding Time Study



Decided to look at three different variables that are suspected of impacting the stability and integrity of a water sample and therefore the holding time;

- Container Type (HDPE vs Glass)
- Amount of sample used (Entire container vs subsample)
- Use of a preservative (Trizma vs no-Trizma/preservative)

Spiked laboratory grade water, in duplicate, at 200 ng/l and collected data at 10 points over 70 days

PFAS Holding Time Study



Spiking groups were

Group 1 – HDPE / 250 mls / No Trizma

Group 2 – HDPE / 250 mls / Trizma

Group 3 – HDPE / 100 mls / No Trizma

Group 4 – HDPE / 100 mls / Trizma

Group 5 – Glass / 250 mls / No Trizma

Group 6 – Glass / 250 mls / Trizma

Group 7 – Glass / 100 mls / No Trizma

Group 8 – Glass / 100 mls / Trizma

Samples pulled from refrigerated storage on a weekly basis starting from Day 0 up until Day 70.

Compounds Studied



Perfluorobutanoic acid
Perfluoropentanoic acid
Perfluorohexanoic acid
Perfluoroheptanoic acid
Perfluorooctanoic acid
Perfluorononanoic acid
Perfluorodecanoic acid
Perfluoroundecanoic acid
Perfluorododecanoic acid
Perfluorotridecanoic acid
Perfluorotetradecanoic acid
Perfluorohexadecanoic acid
Perfluorooctadecanoic acid
N-methylperfluoro-1-octanesulfonamidoacetic acid
N-ethylperfluoro-1-octanesulfonamidoacetic acid

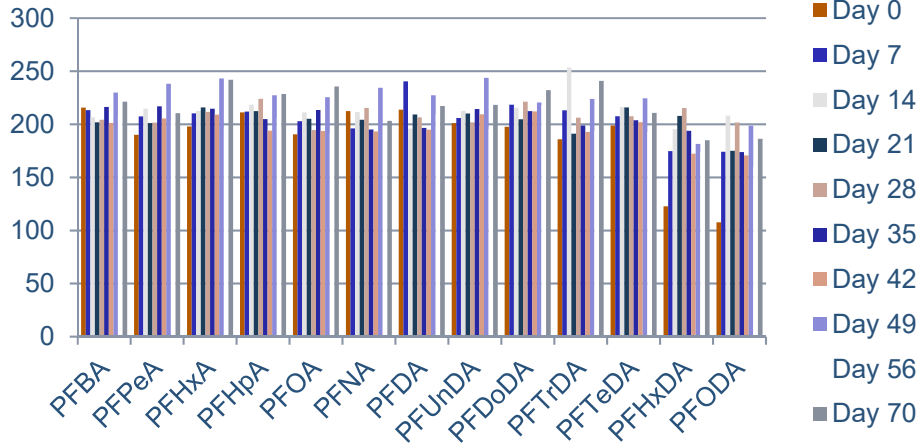
Perfluorobutanesulfonate
Perfluoropentanesulfonate
Perfluorohexanesulfonate
Perfluoroheptanesulfonate
Perfluorooctanesulfonate
Perfluorononanesulfonate
Perfluorodecanesulfonate
Perfluorododecanesulfonate
4:2 Fluorotelomer sulfonate
6:2 Fluorotelomer sulfonate
8:2 Fluorotelomer sulfonate
10:2 Fluorotelomer sulfonate

Compounds in red are the ones listed in EPA 537 ver 1.1

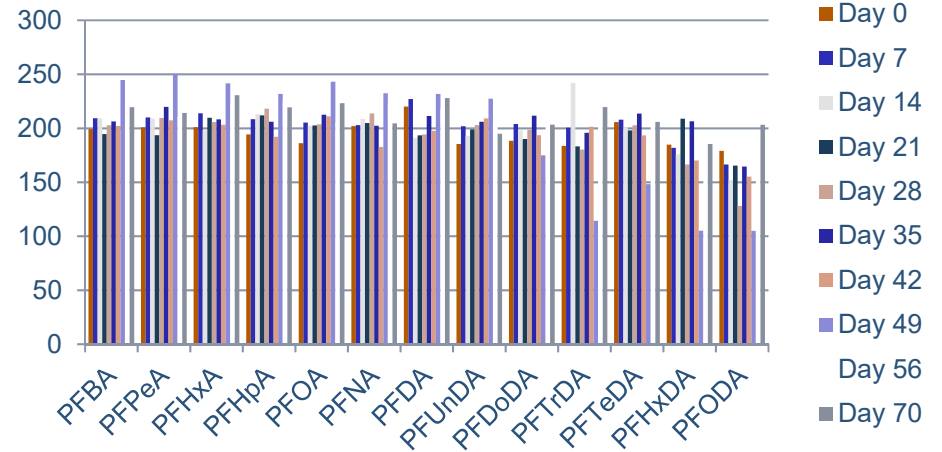
No-Trizma vs Trizma



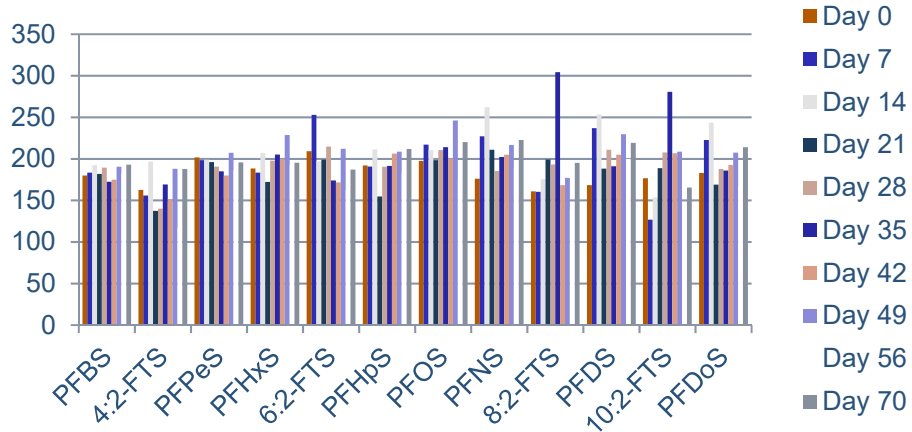
HDPE / 250 / No Trizma



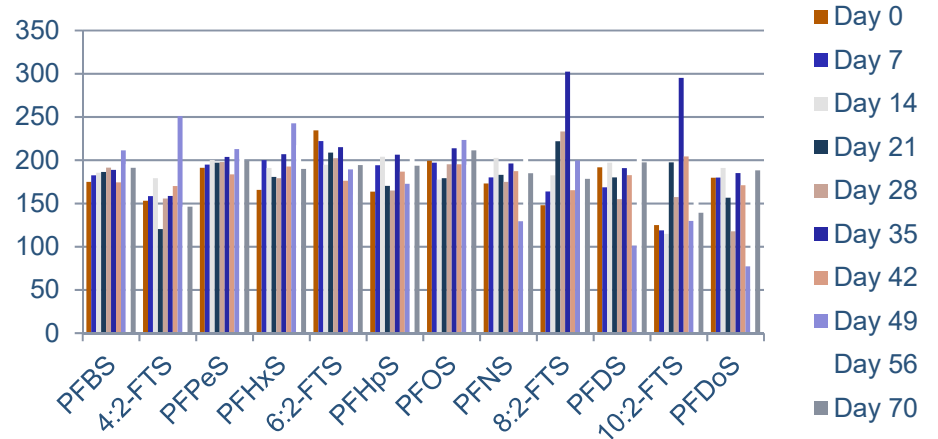
HDPE / 250 / Trizma



HDPE / 250 / No Trizma



HDPE / 250 / Trizma



Observations

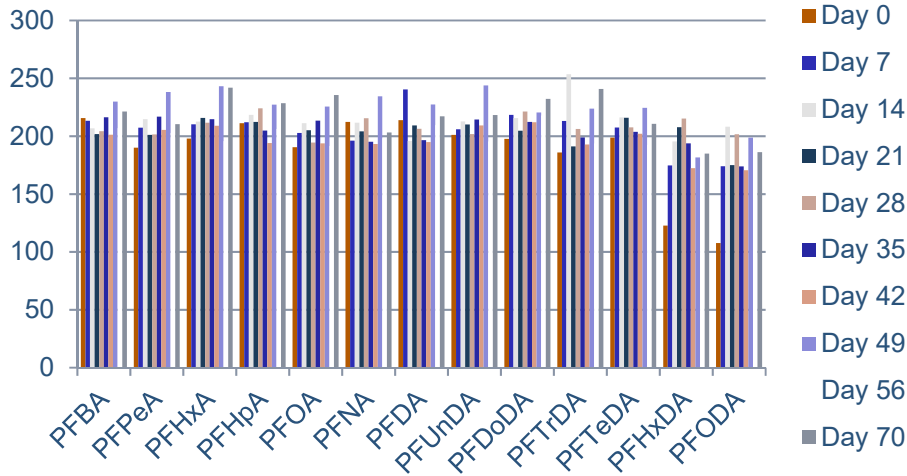


- Little to no difference observed between Trizma preserved and unpreserved containers
- Performance of longest chain length carboxylic acids (C16 and C18) not as solid as shorter chain
- Sulfonates, overall, don't perform as well as carboxylates
- Reasonable good performance for both unpreserved and Trizma preserved out to 70 days

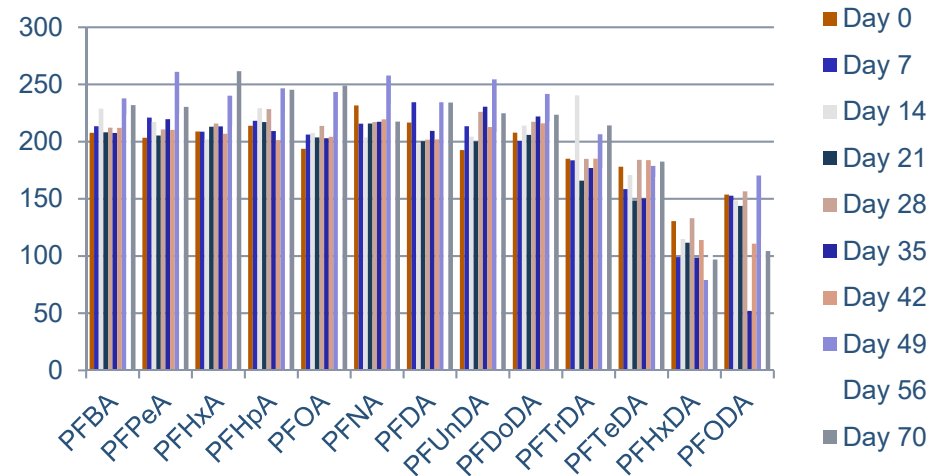
Entire Container vs Subsample



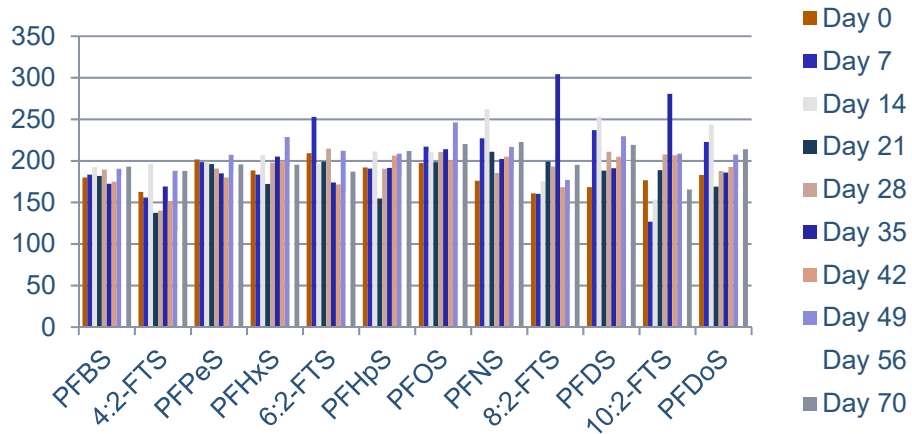
HDPE / 250 / No Trizma



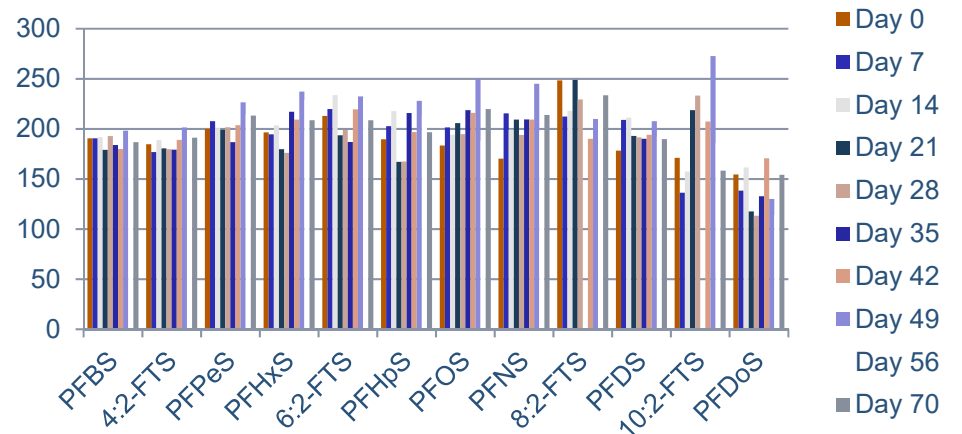
HDPE / 100 / No Trizma



HDPE / 250 / No Trizma



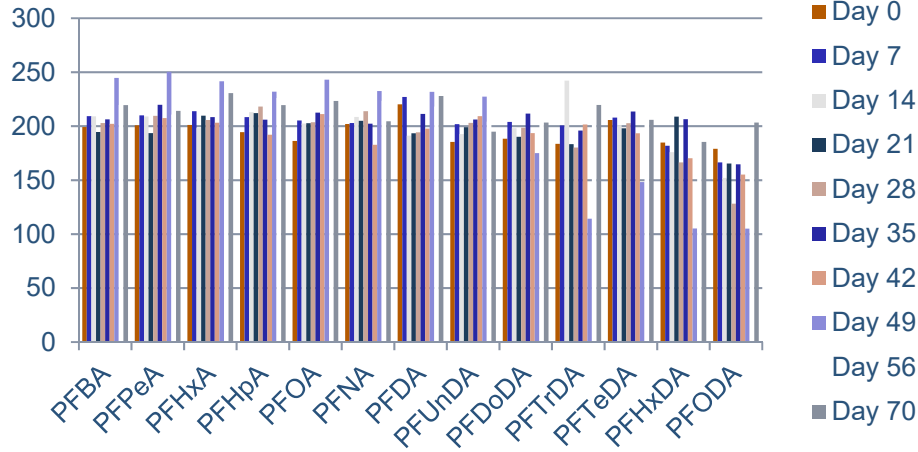
HDPE / 100 / No Trizma



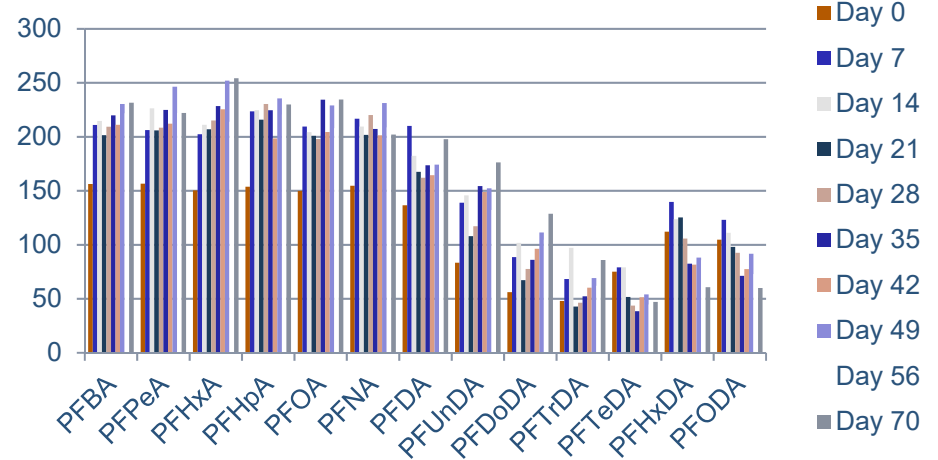
Entire Container vs Subsample



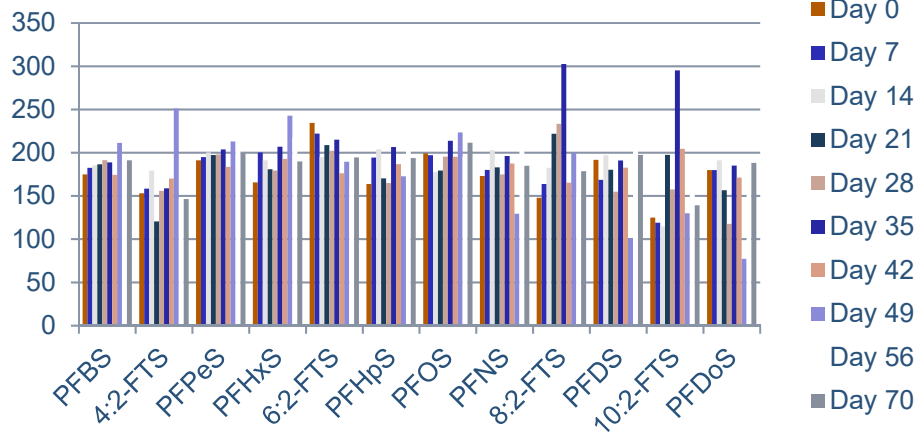
HDPE / 250 / Trizma



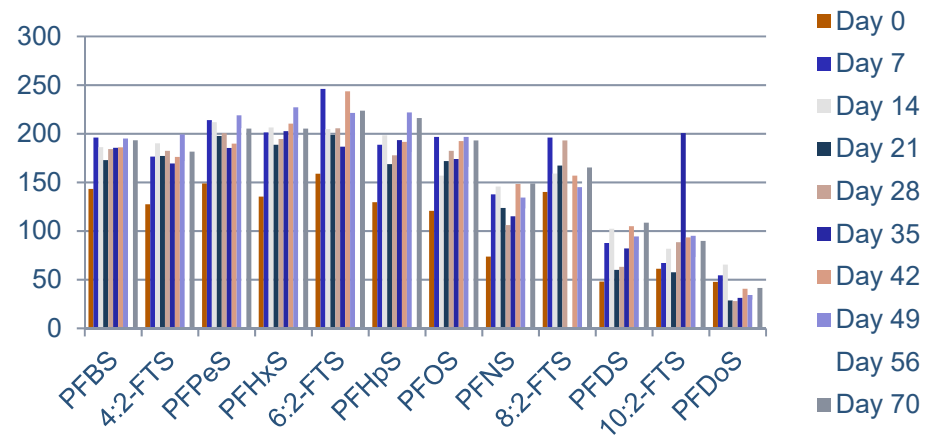
HDPE / 100 / Trizma



HDPE / 250 / Trizma



HDPE / 100 / Trizma



Observations

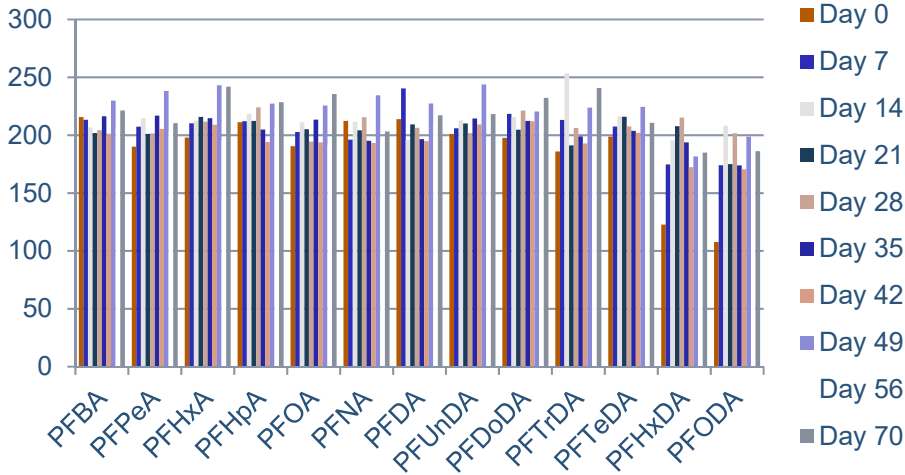


- Presence of Trizma has significant impact on subsampled aliquot for anything past C10 carboxylate and C9 sulfonate
- In unpreserved HDPE container, little impact over 70 day period until C13 carboxylate and C10 sulfonate.
- Implications for evaluation of results from analysis of very high level samples.

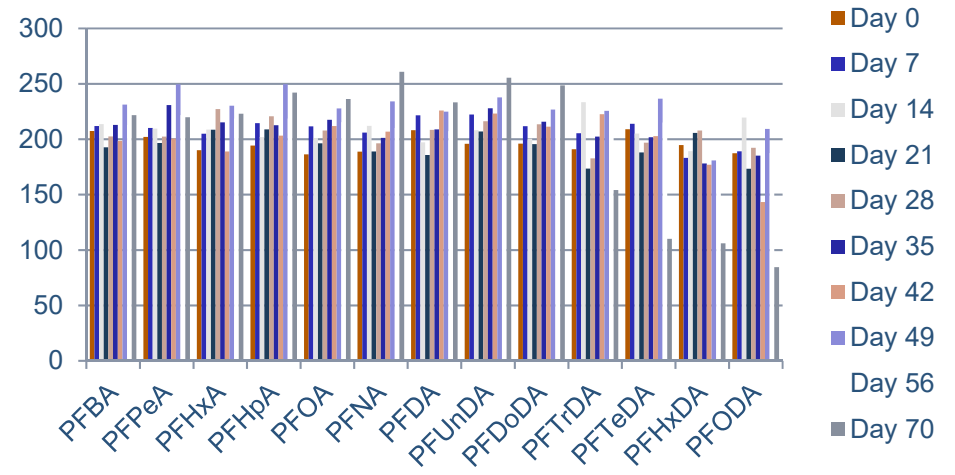
HDPE vs Glass



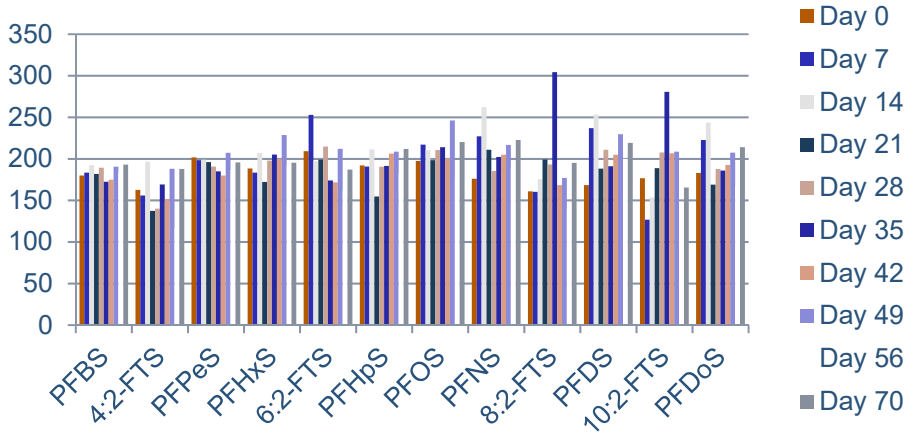
HDPE / 250 / No Trizma



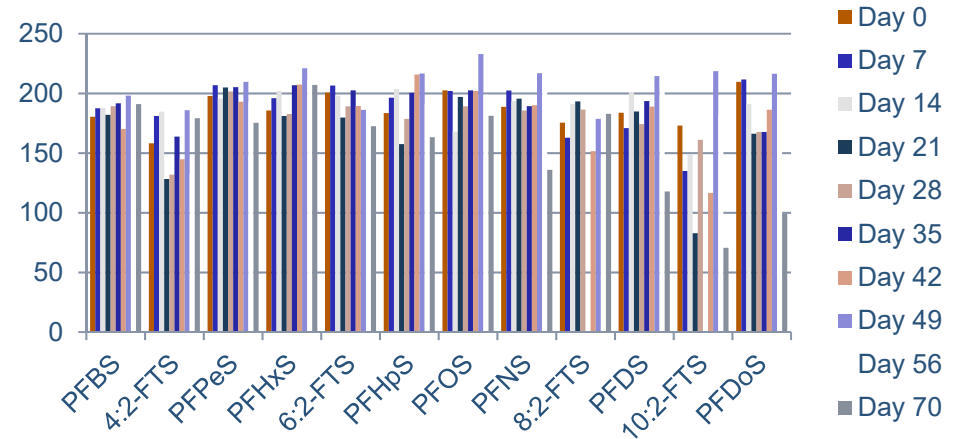
Glass / 250 / No Trizma



HDPE / 250 / No Trizma



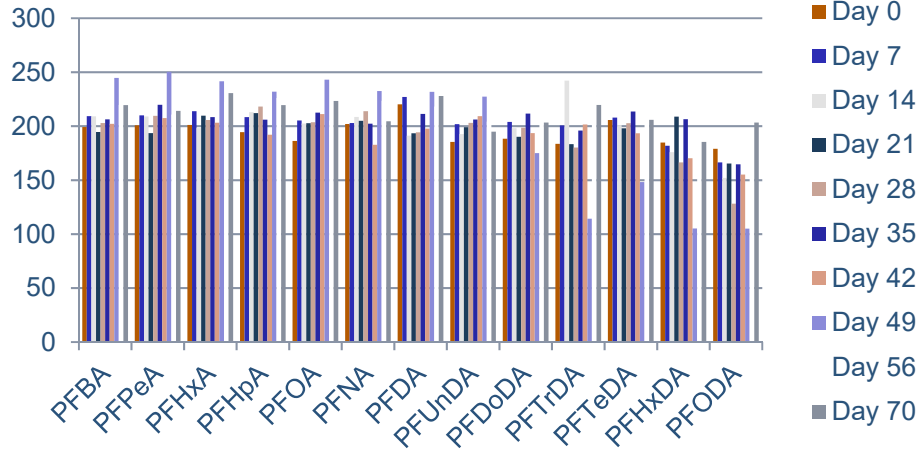
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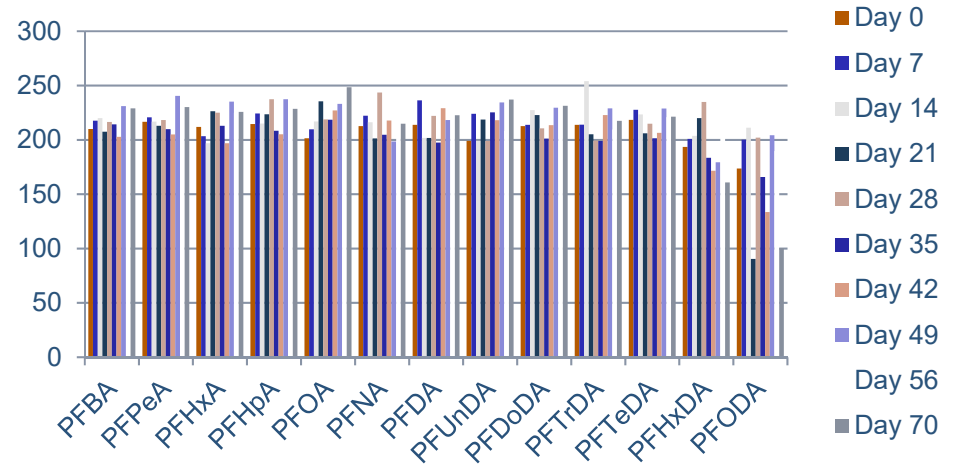
HDPE vs Glass



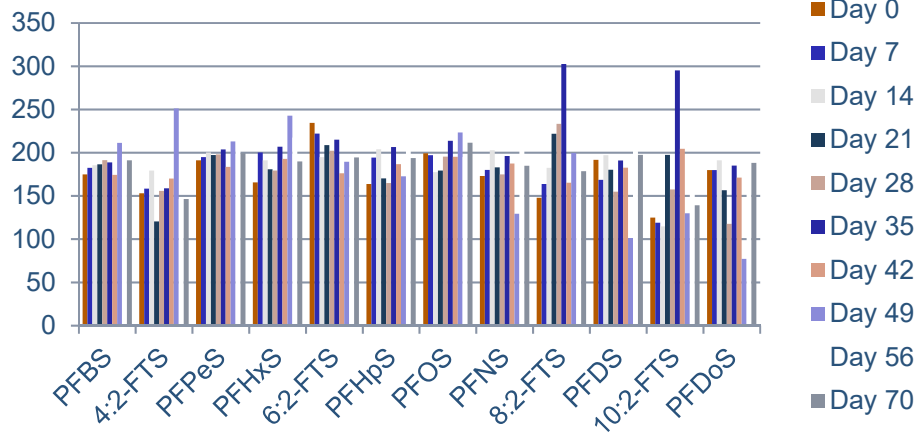
HDPE / 250 / Trizma



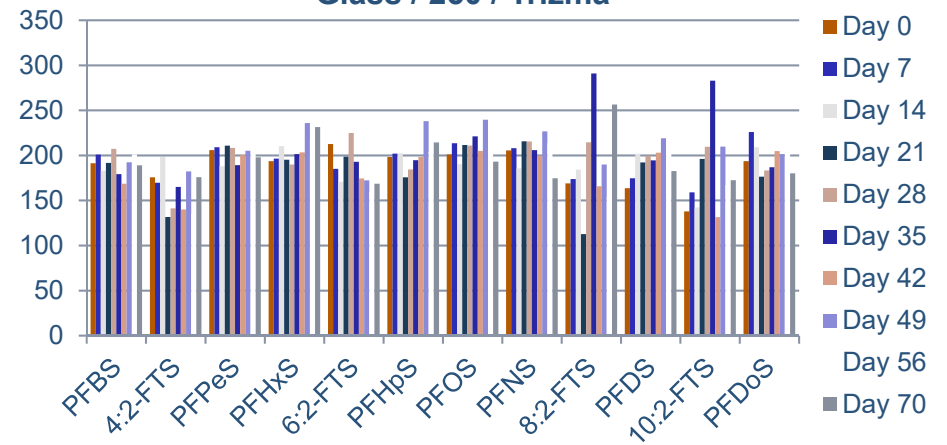
Glass / 250 / Trizma



HDPE / 250 / Trizma



Glass / 250 / Trizma



Observations

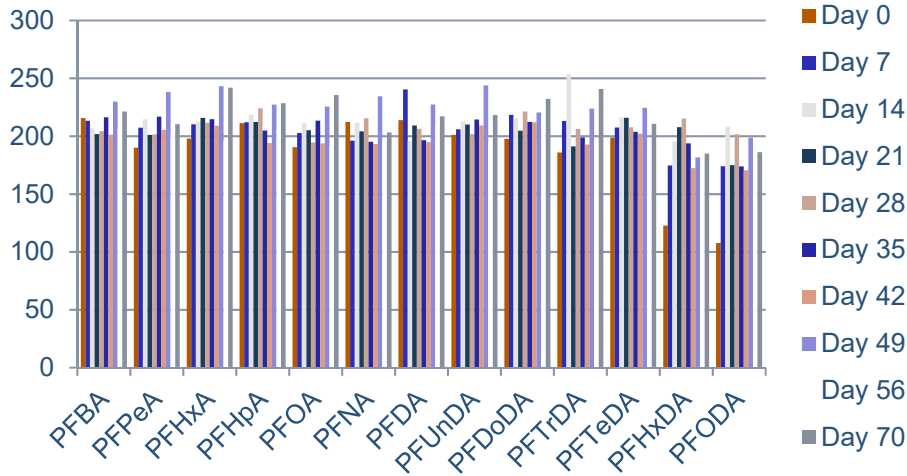


- Glass performed surprisingly well in the unpreserved container as compared to HDPE.
- For the 537 ver 1.1 list of compounds glass looked to perform as well as HDPE.
- Longer chain acids and sulfonates fell off between day 56 and day 70
- Trizma preserved did not seem to contribute any advantage to performance and recovery

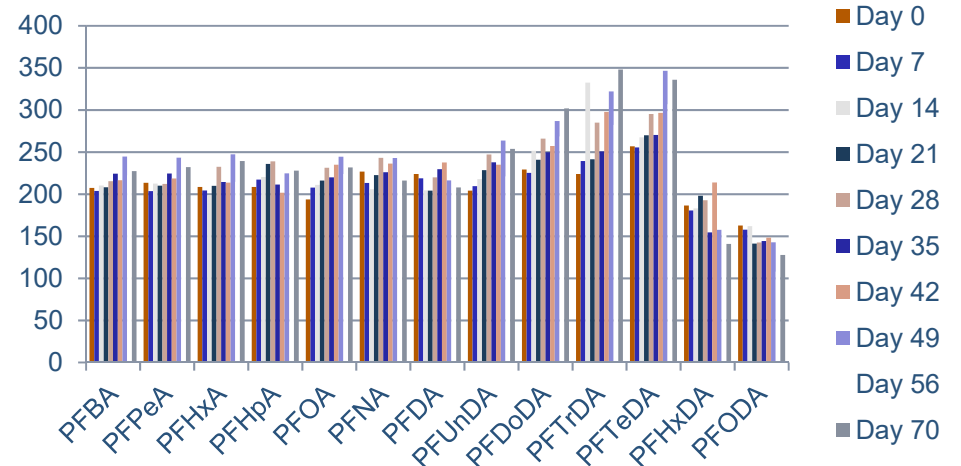
HDPE/Full Container vs Glass/Subsample



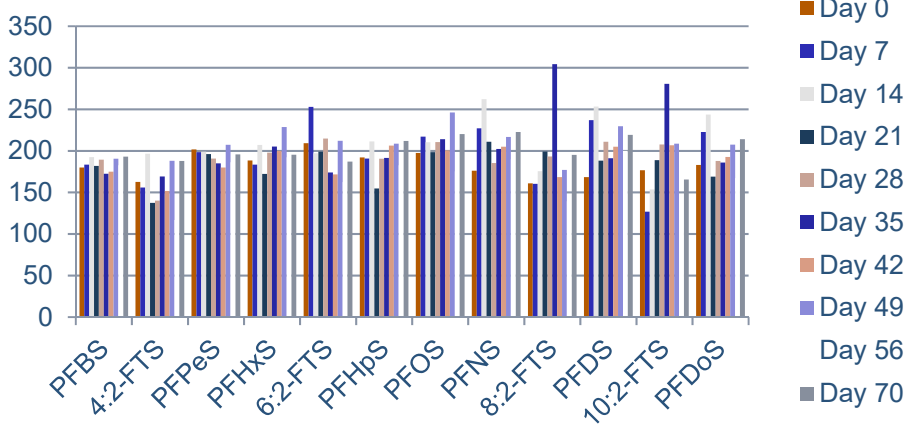
HDPE / 250 / No Trizma



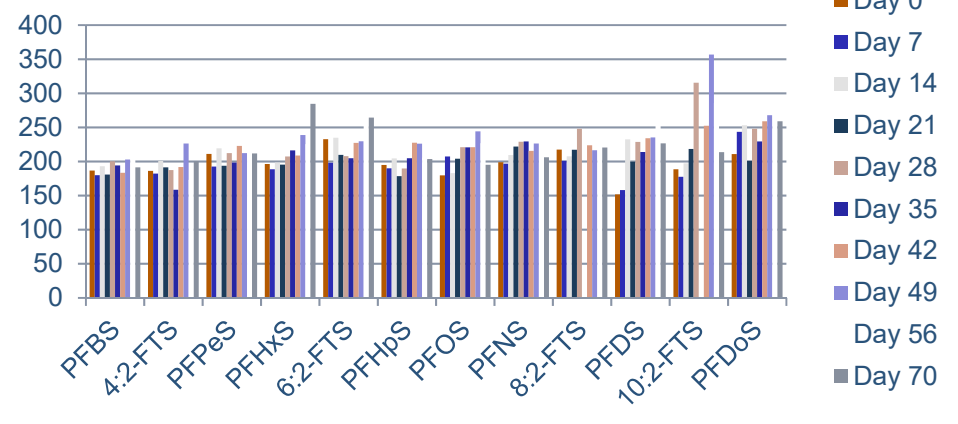
Glass / 100 / No Trizma



HDPE / 250 / No Trizma



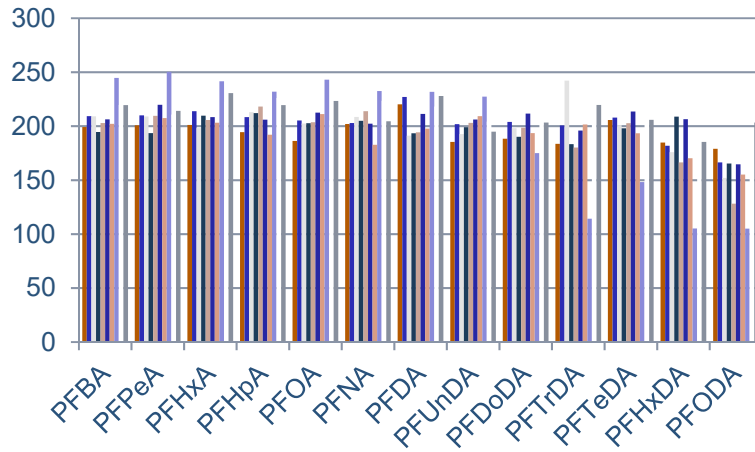
Glass / 100 / No Trizma



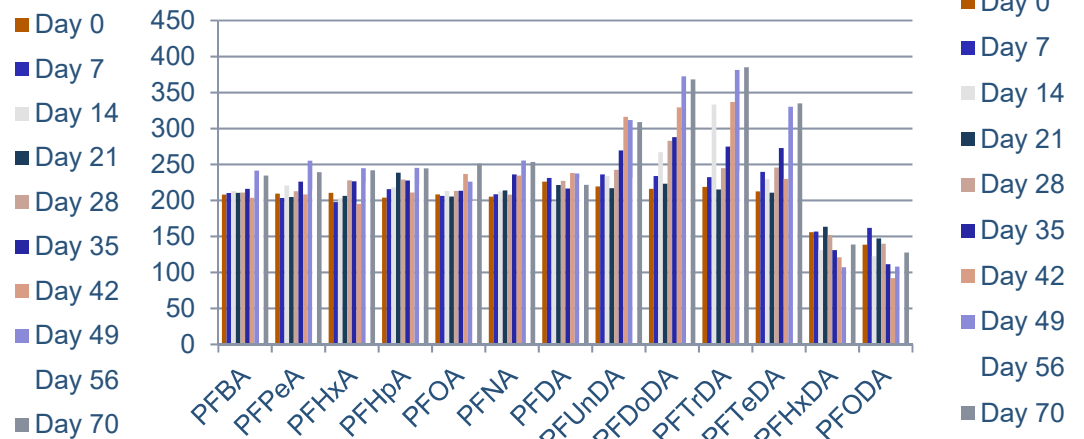
HDPE/Full Container vs Glass/Subsample



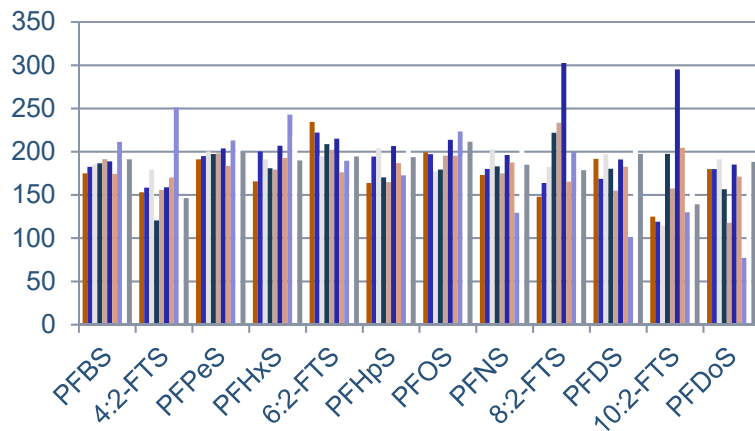
HDPE / 250 / Trizma



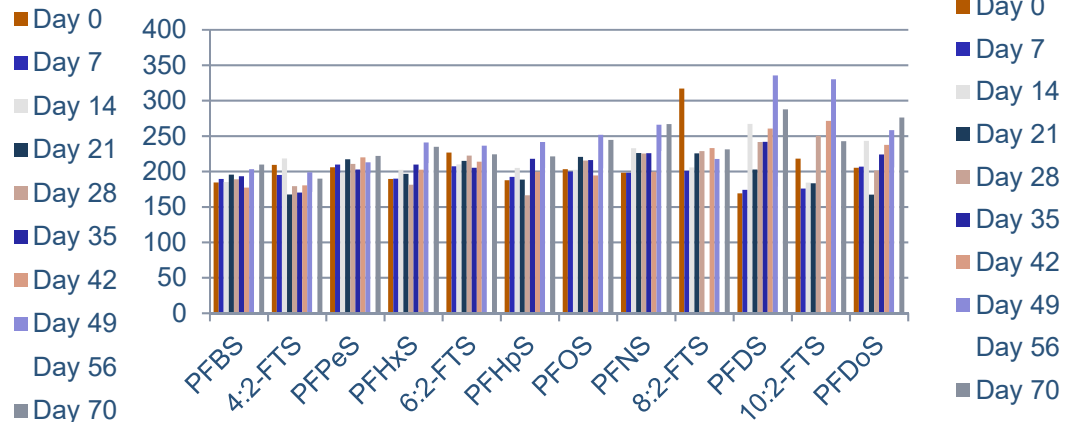
Glass / 100 / Trizma



HDPE / 250 / Trizma



Glass / 100 / Trizma



Observations



- Not much difference between unpreserved and Trizma preserved, HDPE and glass
- Unexplained contamination for longer chain carboxylates (C11-C14) in both unpreserved and Trizma preserved containers from day 35 and later extractions
- C16 and C18 carboxylates significantly impacted in both

Conclusions



- The use of Trizma does not appear to provide any advantages (buffering) to the holding time stability.
- Under certain conditions, glass containers appear to demonstrate reasonable performance. Lid liner would be a concern.
- Whole sample container should be used, except where expected concentrations would be high and impact on overall results negligible.
- Holding times well in excess of 14 days, even for an expanded list of compounds could be used.

Thank you



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